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L7: Entry 3 of 3

File: DWPI

Jan 22, 1975

DERWENT-ACC-NO: 1975-09152W

DERWENT-WEEK: 197506

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TITLE: Coating of water-soluble particles - with aq fluids in a fluidized-bed reactor

PATENT-ASSIGNEE:

ASSIGNEE

CODE

NOVO TERAPEUTISK LAB AS

NOVO

PRIORITY-DATA: 1974GB-0010842 (March 11, 1974), 1973GB-0034993 (July 23, 1973)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
BE 817930 A	January 22, 1975	N/A	000	N/A
AT 7406068 A	January 15, 1980	N/A	000	N/A
CA 1049334 A	February 27, 1979	N/A	000	N/A
CH 604923 A	September 15, 1978	N/A	000	N/A
DE 2435008 A	February 6, 1975	N/A	000	N/A
DK 7403845 A	March 3, 1975	N/A	000	N/A
FI 7402194 A	April 1, 1975	N/A	000	N/A
FR 2238530 A	March 28, 1975	N/A	000	N/A
GB 1483591 A	August 24, 1977	N/A	000	N/A
JP 50071583 A	June 13, 1975	N/A	000	N/A
NL 7409683 A	January 27, 1975	N/A	000	N/A
SE 7409471 A	February 17, 1975	N/A	000	N/A

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no patent
available*

INT-CL (IPC): B01J 2/16; B01J 8/24; B01J 13/02; B05C 0/00; C11D 7/42; C11D 17/06

ABSTRACTED-PUB-NO: BE 817930A

BASIC-ABSTRACT:

In coating of water-soluble or water-dispersible particles by fluidized-bed method, the particles are introduced into the reactor, then the coating fluid, comprising essentially an aq. dispersion or solution of a macromolecular film-former, is sprayed into the reactor, so that the relative humidity of the effluent air is below 100% and that the maximal size of the spray droplets is no greater than the minimal size of the particles to be coated. A very thin coating may be applied in an aq. medium without clumping of the particles. The process may be continuous or batch. Suitable coating agents are e.g. methylcellulose, gelatin, etc.

TITLE-TERMS: COATING WATER SOLUBLE PARTICLE AQUEOUS FLUID BED REACTOR

DERWENT-CLASS: A32 A97 B04 C03 J04 P42

CPI-CODES: A11-B05A; A12-B; A12-W12; B04-B02C; B04-B04A; B04-C02; B04-C03; B12-M11; C04-B02C; C04-B04A; C04-C02; C04-C03; C12-M11; J04-A06;

CHEMICAL-CODES:

Chemical Indexing M1 *01*

Fragmentation Code

V800 V751 V752 V753 V754 V731 V732 V733 V734 V735
V711 V712 V713 V714 V743 F423 H211 J111 J171 H401
H481 J521 H521 H523 H581 H583 H584 H589 H721 M232
M233 M331 M333 N100 M430 M510 M520 M521 M530 M540
M740 M750 M782 R003 R031 R032 R033 R034 R036 R038
R043 M423 M902

Chemical Indexing M1 *02*

Fragmentation Code

V800 V751 V752 V753 V754 V731 V732 V733 V734 V735
V711 V712 V713 V714 V743 F423 H211 J111 J171 H401
H481 J521 H521 H523 H581 H583 H584 H589 H721 M232
M233 M331 M333 N100 M430 M510 M520 M521 M530 M540
M740 M750 M782 R031 R032 R033 R034 R036 R038 R043
M423 M902

Multipunch Codes: 012 028 03- 074 077 081 101 147 198 230 231 24- 240 252 256 315 336 352
397 398 431 432 434 436 445 45- 477 52- 575 583 589 596 603 62& 623 624 645 678 688 720
721

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L16: Entry 1 of 1

File: USPT

Feb 22, 1977

DOCUMENT-IDENTIFIER: US 4009076 A

TITLE: Enzyme granules

BSPR:

The granule core preferably contains a cohesive organic material as binder, the material either melting or dissolving in water at the temperature of use of the granule in order to release the enzyme. Examples of suitable cohesive organic materials are fatty acids containing from 8 to 30 carbon atoms, for instance lauric, palmitic and stearic acids, and fatty acids, coconut oil and tallow; fatty alcohols containing from 12 to 30 carbon atoms, for instance lauryl alcohol and the fatty alcohol derived from tallow; and monoglycerides and diglycerides, for instance 1-monostearin, 1,3-distearin, 1-monopalmitin and 1,3-dimyristin. Preferably the cohesive organic material is a nonionic detergent-active compound, particularly one which has a melting point within the range from 0.degree. to 30.degree. C, especially 15.degree. to 25.degree. C. Suitable nonionic detergent-active compounds are condensates of ethylene oxide with organic compounds having reactive hydrogen atoms, for instance a fatty acid of 8 to 18 carbon atoms, a fatty alcohol of from 8 to 24 carbon atoms, or an alkyl phenol whose alkyl group has from 5 to 18 carbon atoms, using from 5 to 50 mols ethylene oxide per mol of reactive hydrogen-containing compound. A nonionic detergent-active compound prepared by condensation of 9 mols ethylene oxide with 1 mol of a mixture of C.sub.11 to C.sub.15 secondary alkanols is particularly suitable.

DEPR:

On to the granule cores thus produced was atomised an aqueous solution containing 45.5% yellow dextrin and 13.6% glucose, with continued agitation. The resulting granules were then dried for 20 minutes in a fluidised bed with air at 40.degree..

DEPR:

Granule cores were prepared as in Example 1 and given a preliminary coating by atomising on to them in the Lodige mixer a solution of anhydrous citric acid in the same ethylene oxide condensate as was present in the slurry (the solution containing 20% citric acid). The resulting granule cores were placed on a rough disc revolving at 350 rpm within stationary smooth vertical cylindrical walls and an aqueous solution containing 45.5% yellow dextrin and 13.6% glucose and also particles of the finely divided silica alumina composition of Examples 6 and 7 were slowly and simultaneously added to the cores. The granules produced were dried for 20 minutes in a fluidised bed with air at 40.degree..

DEPR:

Enzyme-carrying granule cores were prepared by extruding a plastic mass of a commercial protease concentrate derived from bacillus subtilis, sodium chloride, dextrin and water, through a metal screen to give noodles of 700.mu. diameter. The noodles were dropped on to a revolving rough disc to form spherical particles which are transferred to a fluidised bed drier where they were dried at 40.degree.-60.degree.. The dry granule cores were given a preliminary coating of nonionic detergent-active compound by a step similar to that described in Examples 8 9.

DEPR:

Granular cores prepared in this way were placed in a bed and fluidised with air at 60.degree. and an aqueous solution of 45% yellow dextrin and 13.6% glucose in an amount which was 12% of the granular cores was atomised through a nozzle projecting into the fluidised bed to give granules having a coating of the dried

projecting into the fluidised bed to give granules having a coating of the dried plasticised resin. The granules (7% of which was coating) were then used to prepared detergent composition as in Examples 1 to 3.

DETL:

Sodium dodecylbenzene sulphonate 14 Sodium soap (a mixture of sodium salts of the fatty acids of tallow and 1 coconut oil in proportion 80:20) Anhydrous alkaline sodium silicate 7 (Na.sub.2 O:SiO.sub.2 ratio 1:2) Coconut monoethanolamide 1 Sodium tripolyphosphate 27.5 Sodium sulphate 6.5 Sodium carboxymethylcellulose 0.5 Sodium perborate 25 Water (with perfume and fluorescer) 18.5

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L11: Entry 1 of 1

File: USPT

Dec 30, 1980

DOCUMENT-IDENTIFIER: US 4242219 A

TITLE: Novel enzyme particles and their preparation

BSPR:

Lubricating agents can be added to the enzyme mix to facilitate the mixture of components being divided into particles by mechanical means. Preferably, about 2% to about 7% (advantageously about 3% to about 5%) of lubricating agent is added to the enzyme mix. Examples of lubricating agents which may be used according to the invention are polymeric substances such as polyvinyl alcohol, paraffin oil or wax, stearic acid, and polyvinylpyrrolidone and glycerol also act as a lubricating agent.

BSPR:

To avoid loss of moisture, the resulting particles are coated, preferably with about 0.5% to about 5% depending on the type of coating agent of a coating which may be selected from e.g. a number of water repellent agents, such as paraffin oil or linseed oil, several types of waxes such as earth wax, cocoa fat, candelilla wax, carnauba wax, ceresine wax, lanolin, paraffin wax, beeswax and mixtures thereof. Agglomeration of the particles, which may occur when they are put into bags for transport, may be avoided by further coating of the particles already coated as described above, with an anti-caking agent such as corn starch or other starches, absorbing silicas, such as Aerosil, or talcum powder.

DEPR:

A mixture of 1 part by weight of glycerol, 2 parts by weight of polyvinylpyrrolidone and 2 parts by weight of Berol 80 (ethoxylated C.sub.18 fatty alcohol containing about 80 ethyleneoxide units) was heated until dissolution occurred and the solution was then added to 26 parts by weight of a Maxatase concentrate (commercial enzyme) of about 1.7 MDU/g. Then, 5 parts by weight of titanium dioxide, 15 parts by weight of glucose syrup (starch hydrolysate of about 43 DE), 15 parts by weight of glucose, 33 parts by weight of Amylogum CLS-0473 (an acetylated amylopectin from Avebe, Holland), and about 1 part by weight of water were added thereto and the total moisture content of the resulting mixture was about 7%. The mixture was mixed to a homogeneous paste at a temperature of about 65.degree. C. and was fed to an extruder provided with a perforated plate having orifices of 1 mm. The extruder was further provided with two rotating cutting knives just behind the perforated plate, and with a jacket fed with cooling or heating water, as desired. The paste was extruded at a temperature of about 65.degree. C. and the extruded strings were immediately cut into small particles by the cutting knives. The particles were cooled in a tray and the thus-obtained particles, having cross-sections of about 2 mm and having a (calculated) proteolytic activity of 0.43 MDU/g were coated with 1% by weight of paraffin oil, followed by 3% of corn starch. The enzyme-containing particles thus obtained were deformable under mechanical pressure without disintegration, but under the normal pressure occurring in the lower parts of commonly used bags the particles were hardly deformed for more than a few percent. The elutriation test showed a value of 18 DU.

DEPR:

Starting from the recipe indicated in Example 1 for the enzyme-containing particles, the following fillers were used: 2% of Aerosil 972; 5% and 10% of bentonite; and 5% of penicillin mycelium (obtained from the fermentation broth resulting from penicillin-G fermentations). The particles were coated with 1% of paraffin oil followed by 1% of Aerosil 972. All fillers indicated turned out to be useful, although the particles containing 10% of bentonite tended to be a

little brittle.

DEPR:

The preparation of the particles of Example 1 was repeated except that Amylogum CLS-0473 was replaced by an equal amount of Paselli MD-30 (a starch hydrolysate having a DE of about 30). The particles were coated with 1% of paraffin oil followed by 1% of Aerosil 972.

DEPR:

400 g of a commercially available washing powder containing sodium perborate were added to an amount of enzyme granulate of Example 10 (Sample 5) previously coated with 1% of a 1:1 mixture of glycerol monostearate and paraffin oil, to make the activity 6,000 DU/g and the composition was mixed thoroughly again. Weighed samples of about 20 g taken from the mixture were analyzed for a storage stability test just after mixing and after 0.5, 1, 2 and 3 months storage at 37.degree. C. The samples were tested by dissolving them in synthetic tap water (containing 111 mg/l of CaO, 27.5 mg/l of MgO and 210 mg/l of NaHCO.sub.3, German Hardness 15.degree.), containing sufficient sodium dithionite to neutralize the action of the perborate, and diluting with synthetic tap water to a concentration suitable for testing the activity in Delft Units (cf. British Pat. No. 1,353,317) (series A in the following Table).

DETL:

test (DU)	Elutriation	Filler	Coating 1	Coating 2
+ 2.5% F 2% A 32 2% A 2.5% E + 2.5% F 3% G 18 2% A 0.5% E + 0.5% F 1% A 10 1% A	5% C 0.5% E 3% G 45 2% A 2.5% E			
-- 1% A 80				

A = Aerosil 972 (a hydrophobic silica adsorbent) C = Arbocel C250 (a fine sawdust) D = Bentonite E = Paraffin oil F = Glycerol monostearate G = Corn starch

CLPR:

1. A process for the preparation of enzyme-containing particles which do not, or substantially do not, dust when subjected to external mechanical pressure comprising homogeneously mixing an amount of an enzyme in dry or substantially dry form sufficient to give the desired enzyme activity in the particles when formed, about 20% to about 60% of a hydrophilic organic cohesive compatible with the enzyme selected from the group consisting of starch derivatives, cellulose derivatives, arabic gum, karaya gum and tragacanth, about 10% to about 60% of a building agent suitable for use in enzyme compositions selected from the group consisting of water-soluble carbohydrates of 5 to 12 carbon atoms, up to 70% of which may be replaced by a starch hydrolysate of about 20 to about 70 DE, and an amount of a moisture regulating agent selected from the group consisting of polyols having 2 to 6 carbon atoms and 2 to 4 hydroxy groups attached to different carbon atoms sufficient to maintain a moisture content in the particles formed of from 5% to 15% at ordinary temperatures and ordinary relative humidities and sufficient water to give a moisture content in the particles formed of from 5% to 15%, mechanically dividing the mixture obtained into particles of the desired size, and coating the particles to prevent loss of moisture with about 0.5 to about 5% of a water repellent agent selected from the group consisting of paraffin oil, linseed oil, earth wax, cocoa fat, candelilla wax, carnauba wax, paraffin wax and beeswax, ceresine wax and lanolin and mixtures thereof.

CLPR:

21. The process of claim 1 wherein additionally 2 to 7% by weight of a lubricating agent selected from the group consisting of paraffin oil or wax, stearic acid, polyvinyl alcohol, polyvinylpyrrolidone and glycerol is added.